PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7 : C01B 33/12, B01J 2/16

A1

(11) International Publication Number: WO 00/51939

(43) International Publication Date: 8 September 2000 (08.09.00)

(21) International Application Number: PCT/NO00/00058

(22) International Filing Date: 17 February 2000 (17.02.00)

(30) Priority Data:
19991083 4 March 1999 (04.03.99) NO

(71) Applicant (*for all designated States except US*): ELKEM ASA [NO/NO]; Hoffsveien 65B, N–0377 Oslo (NO).

(72) Inventors; and
(75) Inventors/Applicants (*for US only*): YAMAMOTO, Kunio [JP/JP]; 1–5–8, Shimonagaya, Kounan–Ku, Yokohama–Shi, Kanagawa–Ken (JP). LÆRUM, Ottar [NO/NO]; Kartheia 20A, N–4626 Kristiansand (NO). MESSELT, Egil [NO/NO]; Justlia 10, N–4634 Kristiansand (NO). GITLESTAD, Svein [NO/NO]; Sommerrogata 10, N–4630 Kristiansand (NO).

(74) Agent: VINDENES, Magne; Elkem ASA Patent Department, P.O. Box 8040, Vaagsbygd, N–4675 Kristiansand (NO).

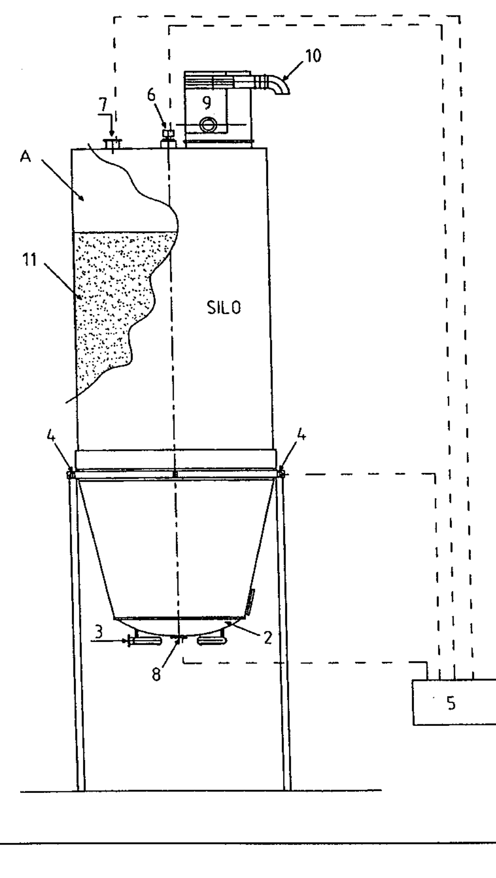
(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(54) Title: MICROSILICA WITH A CLOSELY CONTROLLED BULK DENSITY, METHOD AND APPARATUS FOR PRODUCTION THEREOF

(57) Abstract

The invention relates to microsilica having a closely controlled bulk weight in the range from 425 kg/m^3 to not more than 550 kg/m^3. The invention further relates to a process for production of microsilica with a closely controlled bulk density where microsilica is continuously or substantially continuously charged to, and is continuously or substantially continuously discharged from an enclosed space and where pressurized air is injected from below into the microsilica in the enclosed space at a flow rate that causes fluidization of the microsilica. The weight of microsilica in the enclosed space and the level of microsilica in the enclosed space are continuously recorded and the bulk density of microsilica in the enclosed space is calculated based on the recorded weight of microsilica in the enclosed space and the recorded level of microsilica in the enclosed space. The weight of microsilica charged to the enclosed space is recorded and the discharge of microsilica having a predetermined bulk density from the enclosed space is regulated in order to maintain a preset bulk weight of microsilica in the enclosed space. Finally, the invention relates to a fluidized bed furnace for the production of microsilica with a closely controlled bulk density.



Title of Invention

Microsilica with a closely controlled bulk density, method and apparatus for production thereof.

Technical Field

The present invention relates to the production of microsilica with a closely controlled bulk weight.

Background Art

In the production of materials having a high silicon content such as silicon, ferrosilicon, silicon carbide and other silicon-containing alloys in smelting furnaces, there is generated a substantial amount of silicon monoxide which is converted to silicon dioxide. A similar form of silica is also retrieved from the production of fused oxides such as mullite, zirconia and zircon mullite. The silicon dioxide is in a very fine form and it is normally referred to as microsilica.

Because of the very light nature of microsilica, it does not remain in the smelting process but rather is carried up with the off gases from the smelting process into the furnace flue. Because escaping dust would be an environmental pollutant, it is necessary that the dust be recovered from the smoke from the smelting furnace. Typical dry methods employed in this regard involve bag house filters and the like.

The very fine microsilica recovered which has a typical weight by volume of 150 – 300 kg/m^3 must then be disposed of. Various utilities for microsilica are known such as in refractory ceramics, as a filler in concrete and rubber and as an anti-caking material for fertilizers. When the material is used in these particular applications, it is, of course, usually necessary to transport it to the end user. Because the microsilica is so light and powdery, transportation costs are inordinately high.

From US patent No. 4,126,424 it is known a reversible waterless process for increasing the bulk density of microsilica comprising charging microsilica to an enclosed space and injecting pressurized air from below into the microsilica at a flow rate that causes fluidization for a period of at least five hours in a batch process.

Even though the batch process disclosed in US patent No. 4,126,424 has been in commercial use for more than 20 years it suffers from three main drawbacks. Firstly, the batch process limits the capacity of the process. Secondly, it is difficult to control the final bulk density of the compacted microsilica produced by the process. Thirdly, the known process tends to produce compacted microsilica having a high bulk density in the range of 550 to 700 kg/m^3 which material is difficult to disperse in some end-use applications.

Even though it is mentioned in US patent No. 4,126,424 that the process may be carried out in a continuous way, there is no indications in the patent how such a process should be carried out in order to control the bulk density of the treated microsilica.

Disclosure of Invention

The main object of the present invention is to provide a continuous method for producing microsilica with a closely controlled bulk density.

Accordingly, the present invention relates to a process for production of microsilica with a closely controlled bulk density where microsilica is continuously or substantially continuously supplied to and continuously or substantially continosly discharged from an enclosed space and where pressurized air is injected from below into the microsilica contained in the enclosed space at a flow rate that causes fluidization of the microsilica, said method being characterized in that the weight of microsilica in the enclosed space and the level of microsilica in the enclosed space are continuously

recorded and that the bulk density of microsilica in the enclosed space is calculated based on the recorded weight of microsilica in the enclosed space and the recorded level of microsilica in the enclosed space, that the weight of microsilica charged to the enclosed space is recorded and that the discharge of compacted microsilica from the enclosed space is regulated in order to maintain a preset bulk weight of microsilica in the enclosed space.

According to a preferred embodiment of the present invention the microsilica is discharged from the enclosed space when the bulk weight of the microsilica in the enclosed space is controlled in the range from 425 kg/m^3 to not more than 550 kg/m^3.

According to another preferred embodiment the microsilica is charged to the top part of the enclosed space and is discharged at or near the bottom of the enclosed space.

Preferably the weight of microsilica in the enclosed space is recorded by placing the enclosed space on weighing cells, and by weighing the enclosed space when its empty, whereby the weight of microsilica in the enclosed space easily can be recorded as microsilica is being charged to the enclosed space.

The level of microsilica in the enclosed space is recorded in conventional way by for instance by use of ultrasonic sound measurement devices.

By the present invention it is possible to continuously compact microsilica to a predetermined bulk density.

When starting the process microsilica is charged to the enclosed space and the flow of compressed air through the fluidizing bottom is started. The bulk weight of the microsilica is then monitored by recording the weight of microsilica in the enclosed space and the level of microsilica in the enclosed space. When the bulk weight of the microsilica in the enclosed spaced has

reached a predetermined value one starts to continuously or substantially continuously discharge microsilica from the enclosed space and at the same time starts to continuously or substantially continuously charge further microsilica to the enclosed space where the weight of discharged microsilica over a certain time interval is equal to the weight of microsilica supplied to the enclosed space.

Description of the Drawing

Figure 1 shows a process outline for the process according to the present invention.

On Figure 1 there is shown a silo 1 having a fluidizing bottom 2 and means 3 for supply of fluidizing air to the fluidizing bottom 2. A part *A* of the wall of the silo is removed in the drawing. The silo 1 is placed on load cells 4 in order to record the weight of the silo. The signals from the load cells 4 are registered in a computer 5. On the top of the silo 1 there is arranged a level indicator 6 for recording the level of microsilica in the silo 1. The level indicator is preferably an ultrasonic sound measurement device. The signals from the level indicator are also registered in the computer 5. Based on signals from the weighing cells 4 and the level indicator 6, the bulk density of the microsilica in the silo is calculated at short intervals.

Untreated microsilica having a low bulk density is charged to the silo 1 through an inlet opening 7. The weight on microsilica charged to the silo 1 is recorded in the computer 5. Microsilica having a controlled bulk density is discharged from the silo 1 through a discharge opening 8. The weight of microsilica discharged through the discharge opening 8 is recorded in the computer 5.

Finally, the silo 1 is on its top equipped with a filter 9 and an outlet opening 10 for air from the silo 1. The microsilica removed from the air in the filter 9 is returned to the silo 1.

When the process is started, air is supplied through the air supply pipe 3 to the fluidizing bottom 2 and microsilica is charged to the silo through the microsilica inlet opening 7 until a fluidized bed of microsilica having a preset level 11 is established in the silo. The recording of the weight of the microsilica and the level of microsilica in the silo 1 is started and the bulk density is then calculated and monitored by the computer 5. In an initial period microsilica is not discharged until the calculated bulk density of microsilica in the silo 1 has reached a predetermined value. When this predetermined value for microsilica is reached, discharging of the microsilica with a controlled bulk density through the discharge opening 8 is started and the weight of discharged microsilica is recorded. At the same time further charging of untreated microsilica is started through the microsilica inlet opening 7.

The weight of microsilica charged to the silo is kept at about the same level as the weight of microsilica discharged from the silo. If the calculated bulk weight of microsilica during discharging of microsilica tends to decrease the discharging rate of microsilica is reduced as well as the charging rate of untreated microsilica. If the calculated bulk weight of microsilica during discharging tends to increase the discharging rate of microsilica is increased as well as the charging rate of microsilica. In this way and by controlled variations in the flow of fluidizing air, it is obtained a steady state process with continuous or substantially continuous charging of untreated microsilica and simultanously or substantially continuous discharging of microsilica having a predetermined closely controlled bulk density.

EXAMPLE

Microsilica obtained from the production of silicon in an electric smelting furnace having a bulk density of 250 kg/m^3 was charged to the silo 1 shown in figure 1 through the inlet opening 7. At the same time pressurized air at room temperature was supplied to the fluidizing bottom 2 through the air supply means 3. The flow of air was regulated in order to fluidize the microsilica in

the silo. A starting amount of microsilica was supplied to the silo 1 to obtain a level of microsilica shown at 11. The weight of the silo 1 was measured by means of the weighing cells 4 before supply of microsilica and the measuring of the weight of the silo was continued throughout the test. The level of microsilica was measured by means of the level indicater 6 and when the level 11 of microsilica was lowered further microsilica was supplied to the silo 1. The bulk weight of the microsilica in the silo was calculated in the computer 5 based on the recorded weight of microsilica and the level 11 of microsilica. This calculation was made at very short time intervals. In this example it was the aim to produce microsilica with a bulk density of 450 kg/m^3. Thus when the bulk density of the microsilica in the silo 1 had reached this value, discharging of treated microsilica having a bulk density of 450 kg/m^3 was started through the discharge opening 8. The weight of discharged microsilica was continuously recorded and a weight of microsilica corresponding to the weight of discharged microsilica was charged through the inlet opening 7. The amount of microsilica was discharged according to the calculated bulk weight of microsilica in the silo 1, such that the bulk weight in the silo 1 was kept as close to 450 kg/m^3 as possible. It was obtained a steady state operation to continuously produce microsilica having a bulk density of 450 kg/m^3.

EFFECT OF THE INVENTION

According to the present invention it is possible to easily obtain, under an operation in the continuous, stable state, microsilica having bulk density in the range from 425 kg/m^3 to not more than 550 kg/m^3.

By using the microsilica having the bulk density in the specific range the microsilica can be easily dispersion added homogeneously to the mixtures for various products, and it is possible to reduce the transportation and storing costs of the microsilica.

CLAIMS:

1. Microsilica with a closely controlled bulk density, c h a r a c t e r i z e d i n that the bulk weight is controlled in the range from 425 kg/m^3 to not more than 550 kg/m^3.

2. Process for production of microsilica with a closely controlled bulk density where microsilica is continuously or substantially continuously supplied to and continously or substantially continuously discharged from an enclosed space and where pressurized air is injected from below into the microsilica contained in the enclosed space at a flow rate that causes fluidization of the microsilica, c h a r a c t e r i z e d i n that the weight of microsilica in the enclosed space and the level of microsilica in the enclosed space are continuously recorded and that the bulk density of microsilica in the enclosed space is calculated based on the recorded weight of microsilica in the enclosed space and the recorded level of microsilica in the enclosed space, that the weight of microsilica charged to the enclosed space is recorded and that the discharge of microsilica having a predetermined bulk density from the enclosed space is regulated in order to maintain a preset bulk weight of microsilica in the enclosed space.

3. Process according to claim 1, c h a r a c t e r i z e d i n that microsilica is discharged from the enclosed space when the bulk weight of the microsilica in the enclosed space is controlled in the range from 425 kg/m^3 to not more than 550 kg/m^3.

4. Process according to claim 1, c h a r a c t e r i z e d i n that microsilica is charged to the top part of the enclosed space and is discharged at or near the bottom of the enclosed space.

5. Process according to claim 1, c h a r a c t e r i z e d i n that the weight of microsilica in the enclosed space is recorded by placing the enclosed space on weighing cells.

6. Process according to claim 4, c h a r a c t e r i z e d i n that the calculation of bulk density based on weight of microsilica in the enclosed space and on the level of microsilica in the enclosed space is done by a computer which receives signals from the weighing cells and from a level indicator for microsilica in the enclosed space.

7. A fluidized bed furnace for the production of microsilica with a closely controlled bulk density where microsilica is supplied to an enclosed space and where pressurized air is injected from below into the microsilica at a flow rate that causes fluidization of the microsilica, c h a r a c t e r i z e d i n that said fluidized bed furnace is equipped in its enclosed space with load cells and a level indicator which are connected to a computer.

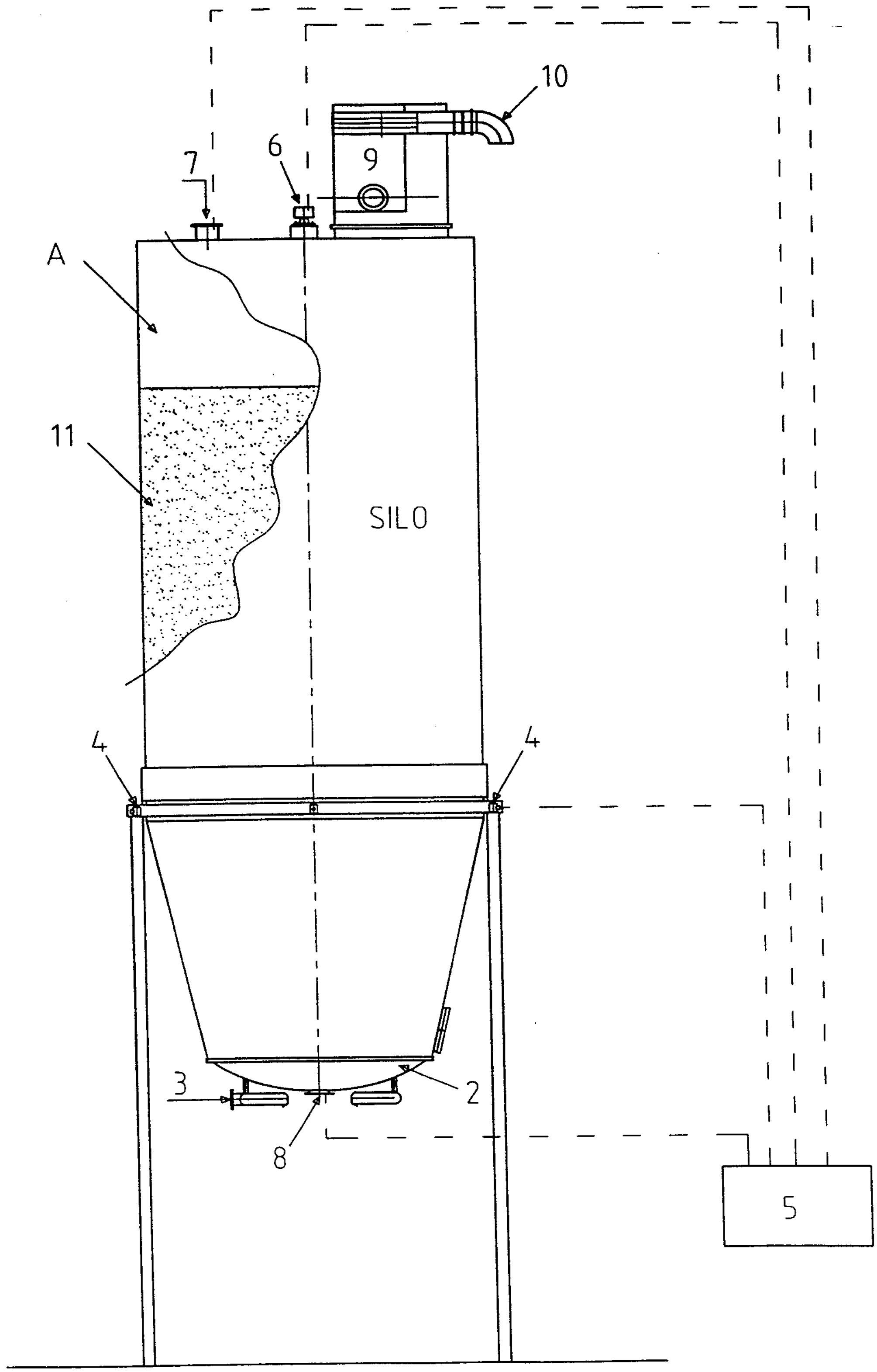


FIGURE 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/NO 00/00058

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: C01B 33/12, B01J 2/16
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: C01B, B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4126424 A (OLE A. KONGSGAARDEN), 21 November 1978 (21.11.78), column 1, line 57 - column 2, line 34, claim 2	1-7
	--	
X	FR 2753110 A1 (PECHINEY ELECTROMETALLURGIE SOCIETE ANONYME), 13 March 1998 (13.03.98)	1
A		2-7
	--	
A	US 5160470 A (STEVEN W. GRAVILLE ET AL.), 3 November 1992 (03.11.92)	1-7
	--	

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
17 July 2000	19 -07- 2000
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer BERTIL DAHL /ipn Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/NO 00/00058

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4126424 A	21/11/78	AR 210396 A	29/07/77
		AU 500946 B	07/06/79
		AU 2456377 A	02/11/78
		BE 853611 A	01/08/77
		BR 7702617 A	16/05/78
		CA 1085140 A	09/09/80
		CH 631419 A	13/08/82
		CS 200512 B	15/09/80
		DE 2717921 A,B,C	03/11/77
		EG 12488 A	31/03/79
		ES 458185 A	16/08/78
		FI 771302 A	28/10/77
		FR 2349539 A,B	25/11/77
		IN 143564 A	24/12/77
		JP 1033227 C	20/02/81
		JP 52131995 A	05/11/77
		JP 55025127 B	03/07/80
		PH 11878 A	11/08/78
		PT 66403 A,B	01/05/77
		RO 72326 A	30/06/81
		SE 7704589 A	28/10/77
		TR 19321 A	28/11/78
		ZA 7702004 A	29/11/78
FR 2753110 A1	13/03/98	NONE	
US 5160470 A	03/11/92	NONE	